



# **Testing and the Challenge of RoHS Compliance**



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**2005 U.S. – China Standards and  
Conformity Assessment Workshop**



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## RESTRICTED SUBSTANCES

- Lead
- Mercury
- Cadmium
- Hexavalent Chromium
- Polybrominated biphenyl (PBB)
- Polybrominated diphenyl ether (PBDE)





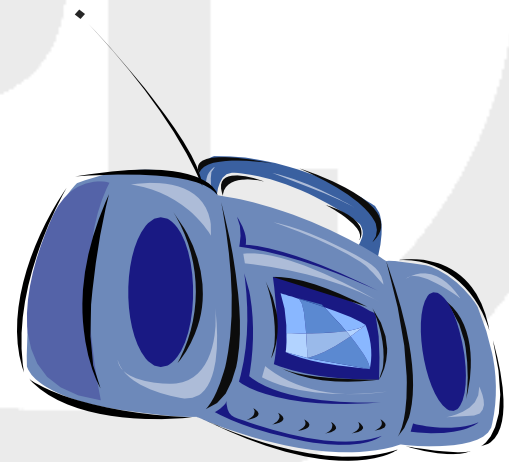
## ‘Homogeneous Material’

- ..cannot be mechanically disjointed into different materials.
- ..of uniform composition throughout“
- ..can be, in principle, separated by mechanical actions such as unscrewing, cutting, crushing, grinding and abrasive processes.

## Maximum Allowable Concentrations

0.01% (100 ppm) Cd

0.1% (1000 ppm) Pb, Hg, PBB, & PBDE





# Electrical & Electronic Equipment Industry Trends



## Now

- Elimination of Lead and Chromate Colorant Systems
- Elimination of Penta and Octa BDE FR Systems
- Increase in Non-Leaded PVC Stabilization
- Increase in Component Testing & Inspection
- Decrease in Self Declaration Acceptance
- Increasing Use of Lead Free Solders
- Increasing Use of Halogen Free Circuit Board Materials
- Increasing Use of Non Hal FR Systems

## Very Near Future

- End Product Design Phase Standards
- Independent Material Certification Schemes



# Tools for Compliance



- ✓ Testing
- ✓ Third Party Certification
- ✓ Supply Chain Management
- ✓ Continuing Regulatory Knowledge
- ✓ Database for Materials or Products
- ✓ Quality Registration Service
- ✓ ECO Labeling
- ✓ Self Declaration



## Traditional Analytical Sample Preparation Techniques

### Acid Decomposition (Pb, Cd, Hg)

- Hot Plate (US EPA 3050B, ASTM E350)
- Microwave (US EPA 3052)
- Kjeldahl Flask (EN-1122)
- Others

### Extraction (PBB/PBDE)

- Organic Solvent (Toluene, etc.)

### Extraction (Cr<sup>+6</sup>)

- Alkaline (US EPA 3060A)
- Boiling Water (ISO 3613)

### Qualitative (Cr<sup>+6</sup>)

- Spot Test (ISO 3613)

### Combustion (Br)

- Oxygen Flask (ASTM D3566, Draft IEC 61189-2C12)
- Oxygen Bomb





## Traditional Analytical Measurement Techniques

- ICP (Pb, Cd, Hg, Cr, Br)
- AA (Pb, Cd, Hg, Cr)
- Direct Mercury Analyzer
- Ion Chromatography (Cr<sup>+6</sup>, Br)
- Titration (Br)
- UV-VIS (Cr<sup>+6</sup>)
- GC-MS (PBB/PBDE Compounds)
- HPLC-MS or UV (PBB/PBDE Compounds)







## Non Destructive Analytical Techniques

- Neutron Activation Analysis (NAA)
- Electron Microscopy (SEM, etc.)
- Proton Induced X-Ray Emission (PIXE)
- X-ray Fluorescence (XRF)



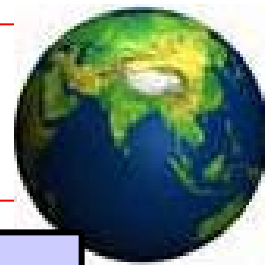
# Traditional vs. XRF Analytical Testing



Aspect	Traditional Analytical Testing	XRF Testing
Capital Instrument Cost	-	Advantage
Speed & Testing Cost	-	Advantage
Accuracy	Advantage	-
Precision	Advantage	-
Sensitivity	Advantage	-
Speciation	Advantage	-
False Negative/Positive Risk	Advantage	-



# Certified Reference Materials (CRM's)



Matrix	CRM Supplier	Catalogue #'s
Polyethylene	Community Bureau of Reference, Belgium	ECR-680 & 681, VDA 001 – 004
Low Alloy Steel	NIST, USA	SRM 2166
Aluminum		SRM 855a, 856a, & 87A
Aluminum	BAM, Germany	CRM-300
Ferro Alloy		CRM-D 502-2
Copper	Community Bureau of Reference, Belgium	CRM075
	Bam, Germany	CRM-211 & BAM 229
Glass	Community Bureau of Reference, Belgium	CRM-664
	BAM, Germany	S004

**More CRM's (e.g. PBB/PBDE in plastic) are needed.**



# Composite vs. Homogenous Material Testing



- Homogeneous Material Testing is Consistent with RoHS Guidelines
- Composite (“Grind it Up”) Material Testing Has High Risk of False Negative or “Pseudo” Compliance

Composite Material	% Mass Contribution	Cadmium Conc. (ppm)	Contribution to Mixed Sample (ppm)
HM Material A	25	120	30
HM Material B	25	60	15
HM Material C	20	0	0
HM Material D	15	0	0
HM Material E	15	4	1
Total Cadmium in Composite Material Sample			46



# Method Detection & Reporting Limits



- **INSTRUMENT DETECTION LIMIT (IDL):** Concentration that produces a signal noise  $>3X$  SD of the mean noise level. The IDL is useful for estimating the constituent concentration or amount in an extract needed to produce a signal to permit calculating an estimated method detection limit.
- **METHOD DETECTION LIMIT (MDL):** Concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is  $>0$ . Determined from analysis of a sample in a given matrix type containing the analyte. The MDL should be determined by multiplying the appropriate one-sided 99% t-statistic by the SD obtained from a minimum of three analyses of a matrix spike or CRM containing the analyte at a concentration 3-5X the estimated MDL.



# Summary

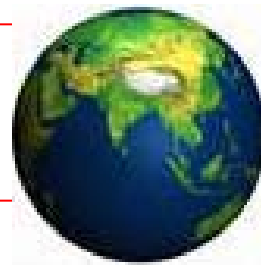


- Testing is an important part of the compliance “toolbox”.
- More CRM’s are needed.
- XRF testing has utility at the top of the supply chain as quick check or “screening tool” with cost and speed advantages.
- Traditional analytical testing has utility throughout the supply chain with accuracy, precision, and sensitivity advantages.
- Composite testing has compliance risks.
- Matrix specific method detection limits are more appropriate than instrument detection limits.



End

Restricted Substances  
Compliance Solutions



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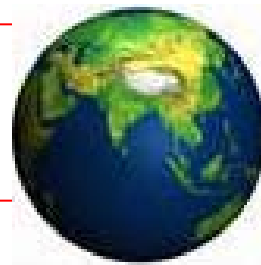
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# Appendix







# X-Ray Fluorescence (XRF) vs. ICP



## XRF vs ICP

Sample	Custom Compounded PVC Material Pb/Cd (ppm)	Lead Content Results		Cadmium Content Results	
		Universal Calibration WDXRF	Hot Plate (EPA 3050B) / ICP	Universal Calibration WDXRF	Hot Plate (EPA 3050B) / ICP
A	0/0	0	6.3	0	0.12
B	26/10	40	27.9	0	10.1
C	261/90	270	279.0	100	99.1
D	353/106	330	365.3	80	93.1
E	366/110	360	389.8	70	108.2
F	991/199	930	1019.7	170	183.2
G	1145/298	1150	1215.7	290	357.1



# X-Ray Fluorescence (XRF) vs. ICP



## XRF vs ICP

Sample	Custom Compounded POF Material Pb/Cd (ppm)	Lead Content Results		Cadmium Content Results	
		Universal Calibration WDXRF	Microwave (EPA 3052) / ICP	Universal Calibration WDXRF	Microwave (EPA 3052) / ICP
A	249/90	180	278.6	0	91.3
B	348/110	180	363.8	0	109.2
C	941/198	570	1093.5	120	194.5
D	1086/296	570	1115.6	160	298.0